*IN THE UNITED STATES PATENT AND TRADEMARK OFFICE*

Applicant: Tadahiro OHMI, et al.

Title: SILICON CARBIDE PRODUCT, METHOD FOR PRODUCING THE SAME, AND METHOD FOR CLEANING SILICON CARBIDE PRODUCT

Appl. No.: 10/566,099

International Filing Date: 7/7/2004

371(c) Date: 5/8/2006

Examiner: Naomi L. Birbach

Art Unit: 1792

Confirmation Number: 9568

**DECLARATION UNDER 37 CFR § 1.132**

The undersigned, Sumio Sano, hereby declares and states as follows:

1) That I am a Japanese national, residing at 11-1-E307, Wada 6-chome, Tamano-shi, Okayama 706-0021. I have been engaged in research related to SiC based material and devices since April 1994. I received my master degree from Division of Electronics and Information Science of Graduate School of Science and Technology at Kyoto Institute of Technology in March 1996. My thesis was focused on studies related to growth of cubic SiC on Si substrate by CVD method. Prior to receiving my graduate study, I was a faculty of Engineering and Design at Department of Electronics and Information Engineering of Kyoto Institute of Technology from April 1990 to March 1994. After completing my master research, I joined Mitsui Engineering & Shipbuilding Co., Ltd in April 1996, and focused my research on manufacturing polycrystalline SiC and designing polycrystalline SiC products. In July 2002, I became a researcher at Tohoku University and Mitsui Engineering & Shipbuilding Co., Ltd, where I have been engaged in research on methods of making high-purity polycrystalline SiC including cleaning methods of polycrystalline SiC.

2) That the following experiments were conducted by myself and/or under my supervision and control.

3) Investigation was made to determine whether or not the cleaning method disclosed in U.S. 2002/0005213 (hereinafter "Otsuki Publication") could reduce a surface concentration of impurity on a surface of polycrystal silicon carbide to less than $1\times10^{11}$ atoms/$cm^2$.

4) The below experiment demonstrates that the cleaning methods taught in Otsuki Publication is inferior to the cleaning method disclosed in the instant application. Specifically, if the cleaning methods taught in Otsuki Publication is used for cleaning the silicon carbide obtained by a CVD method, at least some of the resulting silicon carbide samples would have a surface concentration of metal impurity greater than $1\times10^{11}$(atom/$cm^2$).

SAMPLES AND CONDITIONS

5) Four polycrystalline silicon carbide samples were prepared by a CVD method. The polycrystalline silicon carbide samples had no pores and a theoretical density of 3.2 g/$cm^3$ ,which is different from that of the sintered silicon carbide. All of the samples were immersed into a solution including 1000 ppm of Fe, and then dried.

6) Two of the four polycrystalline silicon carbide samples (Samples A1 and A2) were cleaned by the cleaning method A disclosed in Otsuki Publication, while the remaining two samples (Sample B1 and B2) were cleaned by the cleaning method B disclosed in the instant application.

7) The cleaning method A (i.e., the cleaning method disclosed by Otsuki Publication) was carried out to Samples A1 and A2 in the following manner:

8) Samples A1 and A2 were dipped into an undiluted solution of cyclohexene kept at 50°C for 15 minutes, while ultrasonic vibration was applied. Samples A1 and A2 were then rinsed with pure water, dipped into an aqueous mixture of hydrofluoric acid and nitric acid (10 ml 49% hydrofluoric acid, 10 ml 68% nitric acid, and 200 ml water) for 30 minutes, and subsequently into pure water. The samples were then dried by nitrogen blow.

9) The cleaning method B according to one embodiment of the instant application was carried out to Samples B1 and B2 in the following manner:

10) Samples B1 and B2 were immersed into SPM ( 40 ml 97% sulfuric acid and 10 ml 37% hydrogen peroxide) for 30 minutes and rinsed with pure water. The samples were then dried by nitrogen blow.

## RESULTS

11) The surface concentration of Fe was then measured on each sample by a total reflection x-ray fluorescence analysis (TXRF). The surface Fe concentrations of the samples are shown in Table 1:

Table 1

| Sample | Concentration of Fe Before Cleaning ($x10^{11}$ atoms/$cm^2$) | Concentration of Fe After Cleaning ($x10^{11}$ atoms/$cm^2$) |
|---|---|---|
| A1 | 53224 | 0.91 |
| A2 | 55516 | 1.50 |
| B1 | 41474 | 0.49 |
| B2 | 83266 | 0.85 |

## CONCLUSION

12) As shown above, not all of the resulting polycrystalline silicon carbide samples obtained by the CVD method have a surface concentration of metal impurity of exceeding $1\times10^{11}$ atom/$cm^2$. It shows that the samples cleaned by the cleaning method A (i.e., Otsuki method) can not always reduce the surface concentration of impurity to less than $1.0\times10^{11}$ atoms/$cm^2$. On the other hand, both samples cleaned by the cleaning method B according to one embodiment of the instant application have metal impurity surface concentrations of less than $1\times10^{11}$(atom/$cm^2$). In other words, the cleaning method of Otsuki Publication is inferior to the cleaning method disclosed in the instant application.

13) Further, the Fe surface concentration of the comparative Example 2 of Otsuki Publication (*see* Otsuki Publication, Table 1) is different from that of the above-mentioned Sample A1 and A2, showing that the sintered silicon carbide mentioned in Otsuki Publication is different in properties from the polycrystalline silicon carbide obtained by the CVD method.

14) I hereby declare that all statements made herein of my own knowledge are true, and that all statements made on information and belief are believed to be true; and further that

these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both (18 U.S.C. 1001), and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

March 17, 2010
Date

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